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Modeling and optimization of methanol steam reforming reaction over $Cu/ZnO/Al_2O_3–ZrO_2$ catalyst using a hybrid artificial neural network

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A Hybrid Artificial Neural Network (HANN) model for estimating the activity of a commercial $Cu/ZnO/Al_2O_3–ZrO_2$ catalyst in a laboratory scale methanol steam reforming reactor has been presented. This model is also capable of predicting methanol conversion, selectivity and rate of hydrogen production. In the proposed model, the decay function of heterogeneous catalysts is combined with a feed-forward artificial neural network. To identify the activity of catalyst, a set of 96 data points during 1900 min of operation are obtained from the laboratory scale reactor. From these data, 56 points are selected for training (60%), 20 data points for testing (20%) and the remained ones for validating the developed hybrid network (20%). Results show that the HANN can appreciably predict the activity of the catalyst, and it is also capable of predicting conversion, selectivity and hydrogen production with the AAD% (average absolute deviation) of 1.296, 0.451 and 0.5816%, respectively. Finally by applying the proposed HANN model, process variables i.e. temperature and water to feed ratio are optimized such that by decreasing the activity of the catalyst, the conversion, selectivity and hydrogen production rate can be preserved as equal as the start of run (SOR) values.

Keywords: Methanol, Steam Reforming, Hybrid Artificial Neural Network, Deactivation, Hydrogen

Fuel cells motive electric vehicles and plants for which hydrogen is the key. Recently, consumption of hydrogen as fuel has absorbed attention mainly due to protecting the environment and also sustainable development[1].

In large scale facilities, hydrogen is usually generated from reforming of hydrocarbons such as natural gas, methane or naphtha. But, in small and medium scale systems, it can be generated from water electrolysis. The other way to generate hydrogen for that scale is the steam reforming of methanol.

Methanol steam reforming (MSR) posses some advantages relative to reforming of hydrocarbon such as low steam to carbon ratio, low reforming temperatures (250-300°C), high quality (sulfur<5 ppm), high energy density, the possibility of using a petrol-like infrastructure for distribution, and ease of handling[2-4]. This process is catalytic and endothermic that can generate a product gas containing approximately 75% of H_2 while maintaining a high selectivity towards CO_2. In early decades, comprehensive studies on reaction kinetics and mechanisms of MSR have confirmed that all three reactions (steam reforming, water-gas shift and decomposition) simultaneously occur in this process[2,5].

The Cu-containing catalysts ($Cu/ZnO/Al_2O_3$) have showed particularly high activity and selectivity to the MSR. Unfortunately, a major concern in using these kind of catalysts is the deactivation phenomenon caused by sintering of Cu particles in initial hours of runs at which reduces the selectivity and conversion of the process[5,6]. Loss of catalytic surface area due to the crystallite growth in the catalytic phase (Cu-particles), loss of support area due to the support collapse, and loss of catalytic surface area due to the pore collapse on metal crystallite are typically known as sintering phenomenon[7]. Similar to other chemical processes, optimal operation is required to guarantee profitability, and therefore, it necessitates using process models. A well developed model is applicable to monitor, simulate and optimize of MSR reaction over $Cu/ZnO/Al_2O_3–ZrO_2$ catalyst, and also it can be a beneficial tool for predicting age of catalyst and designing compressed fuel processors.

Except to kinetic-based models, classified as deterministic or first principal models, the use of an artificial neural network (ANN) can be practical.

ANNs are high performance non-linear analytical tools which are capable of establishing the relationship between the input/output data without prior knowledge of the correlation between the variables involved in the system[8]. They are a simple alternative for processes that involve phenomena with complex and difficult mathematical description[9]. In some cases, they provide better results than empirical correlations[10]. Additionally, ANNs can be applied as hybrid models. The term hybrid modeling is used to describe the incorporation of prior knowledge about the process under consideration in a neural network modeling approach[11,12]. Up to now, ANN models have been applied for modeling of many refining processes such as hydro desulfurization, hydrocracking, delayed coking, catalytic reforming and thermal cracking[13-21]. Furthermore, ANN is an efficient tool to model the sophisticated relationship between input and output process variables in catalytic–dielectric barrier discharge plasma[22-24], three phase fluidized beds[25] and liquid membrane systems[26]. Additionally, in recent works, hybrid-ANN models with the combination of the decay function of heterogeneous catalysts and a recurrent-layer artificial neural network were applied to predict the activity of the catalyst in industrial scales light naphtha isomerization[27] and heavy naphtha reforming plants[28].

In the previous work, we studied the kinetic of MSR reactions[29]. In this research, a hybrid-ANN model is proposed which combines the decay function of heterogeneous catalysts with a feed-forward artificial neural network to model, monitor the catalyst activity, predict the age of catalyst and optimize the process variables of steam reforming reaction over a commercial $Cu/ZnO/Al_2O_3–ZrO_2$ catalyst.

Experimental Section

Pilot plant device

A schematic diagram of the experimental equipment is demonstrated in Fig. 1 which consists of feed, reaction and analysis section. The gaseous part of the feed is composed of H_2, CO_2 and carrier gas N_2.

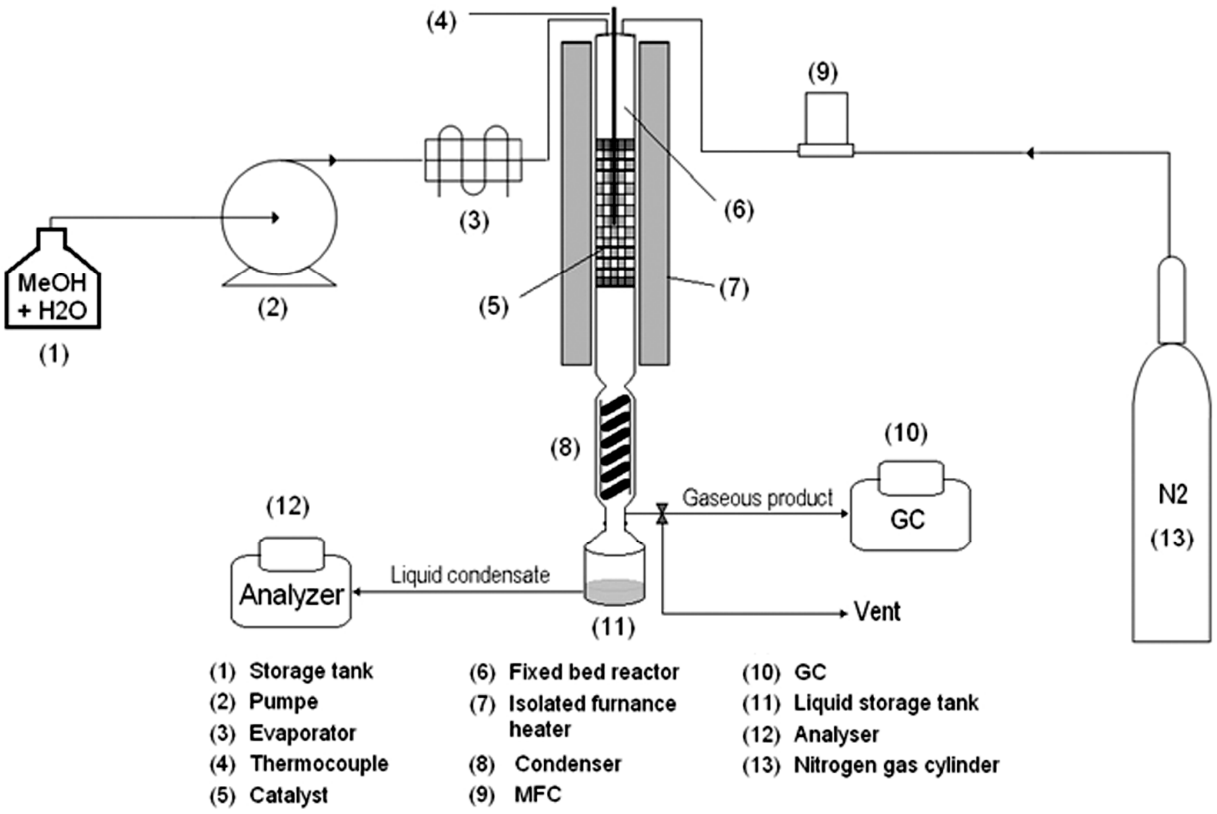

Fig. 1 — Experimental device for steam reforming over $Cu/ZnO/Al_2O_3–ZrO_2$ Catalyst: 1.Storage tank (Deionized water and 99.7% MeOH), 2.Pump, 3.Evaporator, 4.Thermocouple module (J Type), 5.Catalyst bed, 6.Tubular packed bed reactor (ASTM 316), 7.Isolated furnance heater, 8.Condensor, 9.Mass flow meter (Bronkhorst), 10.Gas chromatograph for gas outlet analysis (Philips Pu-4600), 11.Storage tank for collecting unconverted water and methanol, 12.Columns for methanol and water analysis(Shimadzue), 13.Carrier gas (99.999% N_2).

Moreover, H_2O and CH_3OH are introduced as liquid feed to the reactor. After reducing the pressure, the mass flow rate of each gas is controlled at the desired value using a mass flow controller (MFC). Also, liquids are transferred from storage tanks by means of diaphragm pumps. After mixing, the feed is entered into the preheating–evaporating and reaction section. The length and internal diameter of this section are 89 cm and 1.8 cm, respectively. Five thermocouples are provided to monitor and control the axial temperature of that. After condensing of steam and methanol, and drying the gas, the effluent gas and liquid were sent to the analyzing section which consists of gas chromatographs (GCs, Shimadzu and HP 5890 for liquid and gas, respectively). These GCs are equipped with thermal conductivity detector (TCD), and uses helium as carrier gas to analyze the methanol, water, H_2, CO_2 and N_2.

Catalyst

To conduct this study, a commercial $Cu/ZnO/Al_2O_3$ catalyst (MSR catalyst) with the cylindrical shape was provided. The physical properties of that are listed in Table 1. To eliminate internal diffusion resistance, 8.2 g of catalyst was ground and sieved (14-20 mesh size), and then it was mixed with 8.2 g of inert α-Alumina (20-25 mesh size). Then, the catalyst was activated according to the following procedure: (i) the catalyst was heated up to a fairly uniform temperature of 170-180°C with the ramp of 50°C/h, and then, N_2 with 0.5-1.0 vol.% of reducing gas (H_2) was introduced to the reactor, (ii) while maintaining the inlet temperature of 175°C, H_2 concentration was raised to 1.5-2 vol.% making sure that the bed temperature did not exceed 220°C; (iii) after reaching to a steady bed temperature, and sensing H_2 purity about 90% of input value, the feed temperature was raised to 205°C, and (iv) H_2 concentration was raised stepwise to 10-15 vol.% to ensure complete reduction of the catalyst.

Table 1 — Physical property of the commercial steam reforming catalyst

Specifications	Values
Bulk density	1500-1600 kg/m^3
Surface area (based on BET analysis)	60-80 m^2/g
Pore volume (based on N_2 isotherm analysis	0.2-0.25 mL/g
Crush strength	35-45 N/tablet
CuO wt%	50%
ZnO wt%	30%
Al_2O_3-ZrO_2 wt%	20%

Modeling methodology

Fundamentals of artificial neural network

ANN is a parallel structure composed of nonlinear nodes which are connected by fixed weights and variables. Since these weights are not related to any physical identities, this approach can be classified as a black-box model. ANN is an information-processing paradigm that is inspired by the way the biological nervous system, such as the brain, processes information[30]. The most common for chemical engineering applications is MLP (multi-layer perceptron), which is a feed-forward neural network[31]. In a feed-forward MLP, neurons consist of at least three layers of nodes including input, output and one or more hidden layers (Fig. 2). For this topology, the information propagates in only the forward direction.

The most widely employed networks have one hidden layer only[32]. Each node within a given layer is connected to all of the nodes of the previous layer. This node sums up the weighted inputs and a bias, and passes the result through a linear function as follows[33]:

$$a_j = \sum_{i=0}^{m} w_{ji} x_i + b_j \qquad ...(1)$$

where w_{ji} is the weight that goes from the input (i) to the hidden neuron (j); b is the bias to the node, and x_i is the input unit of the neuron. By utilizing an activation function (f), the output of the neuron can be written as follows:

$$z_j = f(a_j) \qquad ...(2)$$

This activation function is applied to model nonlinear behavior of the process. In this work, the

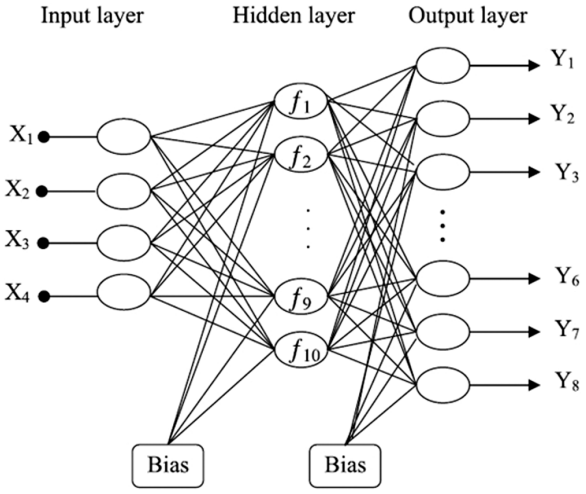

Fig. 2 — Schematic diagram of a three layer ANN[16]

activation function utilized for the hidden and output nodes is the tangent sigmoid function as follows:

$$f(a) = \frac{e^{a} - e^{-a}}{e^{a} + e^{-a}} = \tan sig(a) \quad ...(3)$$

After creating the structure of the ANN, training procedure is carried out by introducing a set of known inputs and outputs. Now, ANN can learn the trend of these data by manipulating the weights and biases, using backward propagation i.e., iterative reduction in training errors using a generalized delta rule[34,35]. So, fitting parameters are adjusted to attain a minimum value for the following mean square error (MSE):

$$MSE = \frac{1}{N}\sum_{k=1}^{N}(Y_{k,actual} - Y_{k,\mathrm{mod}el})^{2} \quad ...(4)$$

where N is the total number of known data; k is the output values; actual refers to the measured outputs from the MSR plant, and model refers to the simulated values by the model.

Hybrid-MLP (HMLP) neural network applied to MSR reaction

The success in obtaining a reliable and robust network depends strongly on the choice of process variables involved, as well as the available sets of data and the domain used for training purposes[36]. Therefore, it is essential to include all momentous variables, affecting the yield and quality of the product, in the input layer of the ANN structure. The input neurons of the developed HMLP for the target MSR unit consists of the minutes on stream (MOS), temperature of the reactor (TR), water to feed ratio (W/F), and CO_2 and H_2 fraction of the feed (CO2F and H2F, respectively) (Fig. 2). The output layer consists of methanol conversion (Conv), selectivity (Sel), and the rate of hydrogen production (FH2).

In the proposed hybrid model, the following exponential law is applied for estimating the activity of the catalyst[37]:

$$\mathrm{a_c} = exp(-\alpha \times \mathrm{t_c}) \quad ...(5)$$

$$t_c = \frac{\sum_{s=0}^{life}(m_f \times t_s)}{W_{cat}} \quad ...(6)$$

where a_c is the activity of the catalyst; α is the decay constant which is dependent to the type of the catalyst; life is the MOS for the current test run; t_s is the time interval between the current test run and the previous one; m_f is the mass flow rate of feed; W_{cat} is the total weight of catalyst in the reactor, and t_c is a dimensionless term, called accumulated feed, representing the total amount of feed passed through the total catalyst weight at a specific life time.

This hybrid-ANN (HANN) model was implemented in MATLAB 2013 environment. A feed-forward neural network consisting of 5 neurons in the input layer and 3 neurons in the output layer was built. The transfer or activation function used in the hidden and output nodes was the tangent sigmoid function. Training of the ANN was carried out using 'trainlm' syntax that applied Levenberg-Marquardt optimization method to estimate weights and biases. Training was performed until finding the minimum MSE between the simulated and actual output variables.

Optimization of MSR plant

The conversion, selectivity and the rate of hydrogen product are identified as the significant output variables of the MSR plant. Therefore, they are maximised simultaneously using the validated HANN model by manipulating the process variables i.e., reactor temperature, water to feed ratio, and CO_2 and H_2 content of the feed.

Although there are many numerical methods that have been presented to solve an optimization problem, in this paper the genetic algorithm (GA) has been chosen. GA is a part of soft computing, a branch of computer science that deals with exploring the search space, selecting the best solution, and working for global optimization[38]. These algorithms (GAs) have been applied to a variety of function optimization problems, and were shown to be highly effective in searching large and complex response surfaces even in the presence of difficulties, such as high dimensionality, multimodality and discontinuity[39]. The GA operates on a population of potential solutions, using the principle of survival of the fittest to produce successively better solutions to a problem. At each generation of a GA, a new set of answers is created by the process of selecting individuals according to their level of fitness in the problem domain and regenerating them using operators mimicked from natural genetics. This process leads to the evolution of populations of individuals that are better suited to their environment than the individuals from which they were created, just as in natural adaptation.

In this research, to optimize the conversion, selectivity and the rate of hydrogen product, the

genetic algorithm function ('gamultiobj') of MATLAB 2013 is applied. This function uses a controlled elitist genetic algorithm which always favors individuals with better fitness values[40]. A controlled elitist GA also favors individuals that can help increase the diversity of the population even if they have a lower fitness value. It is important to maintain the diversity of population for convergence to an optimal Pareto front. Diversity is maintained by controlling the elite members of the population as the algorithm progresses.

Results and Discussion

To determine the activity of the commercial MSR catalyst, a set of 96 data points during 1900 min were gathered from the laboratory scale experiments. From these data points, 56 data points were selected for training (60%), 20 data points for testing (20%) and the remained ones for validating the HANN model (20%).

At first, it was assumed that the catalyst was not deactivated during its life and the decay constant (α in Eq. 5) was zero. So, the activity of the catalyst did not affect the conversion of reaction for all data points. To identify the optimum number of neurons in the hidden layer, the value of MSE with different numbers of nodes were calculated. After 50,000 iterations, it was found that the network with 8 hidden nodes had the least MSE and increasing the number of hidden nodes did not considerably improve the MSE of the trained HANN. Therefore, to prevent over-fitting, 8 hidden nodes were selected. After training network, it was found that the best HANN to predict the conversion of MSR had the MSE of 0.297. Now, to improve the accuracy of the model, the decay constant was manipulated from 0 to 0.00002 with the step size of 1E-6, and the HANN was retrained. After doing this procedure, it was found that the HANN model with 8 hidden nodes and a decay constant of 7E-6 can predict the conversion with the MSE of 0.2042.

In Fig. 3, the activity versus MOS using the estimated decay constant (7E-6) is shown. From this figure, it is discovered that the target commercial $Cu/ZnO/Al_2O_3$ catalyst sharply deactivates such that it loses about 22 percent of its activity after 1900 min of starting the reaction. As discussed later, losing the activity should be rectified by manipulating the other process variables, especially reaction temperature to sustain the conversion, selectivity and production rate at the desirable values.

Using the decay constant of 7E-6, HANN model was trained, tested and validated to simulate the other output variables of the MSR plant. A comparison between the predicted and measured values of selectivity and rate of hydrogen production are presented in Fig. 4 and 5, respectively. As seen, this model can appreciably simulate these output variables with a high accuracy. Moreover, AAD% and MSE of prediction are presented in Table 2. It is obvious that the developed hybrid model is reliable enough to be applied for predicting output variables of the target plant.

Now, by applying the HANN model, the operational variables of the reaction i.e. TR, and W/F were optimized to yield the highest conversion, selectivity, and hydrogen production rate, simultaneously. After that, it was tried to compensate

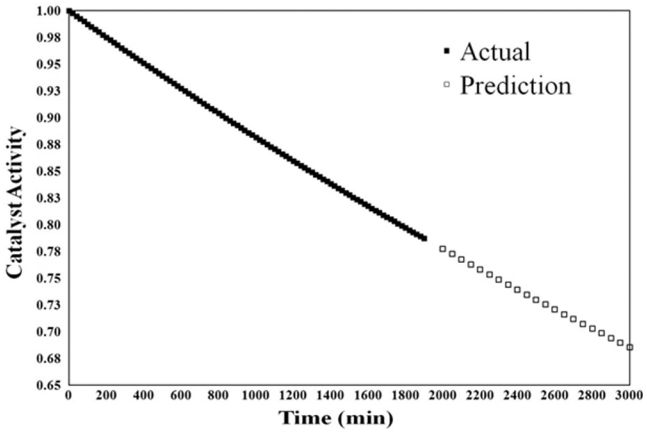

Fig. 3 — Activity of the methanol reforming catalyst vs. time on stream estimated by HANN model

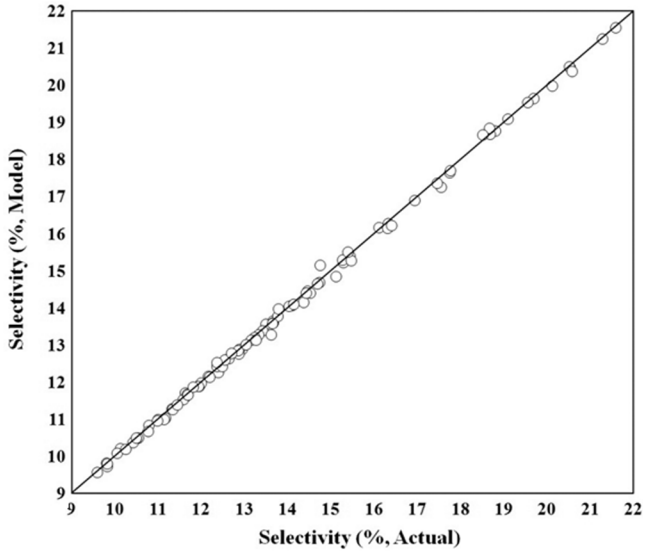

Fig. 4 — Selectivity predicted by the HANN model vs. actual values

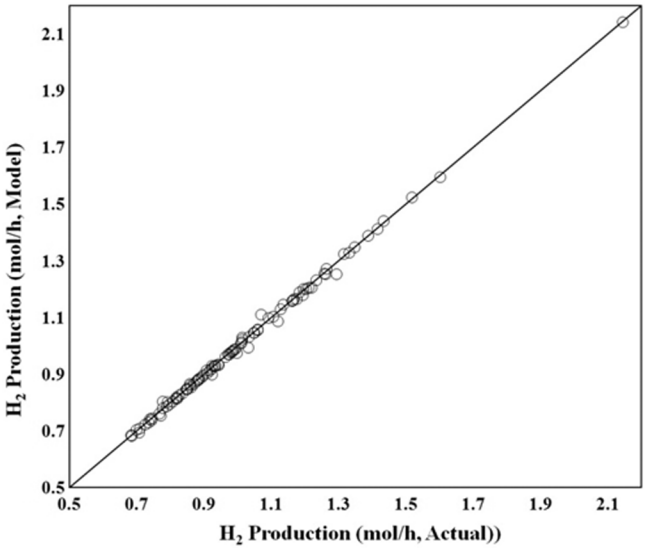

Fig. 5 — Rate of hydrogen product predicted by the HANN model vs. actual values

Table 2 — MSE and AAD% of prediction for output variables of methanol steam reforming reaction model

Variable	MSE	AAD%
Conversion (%)	0.2042	1.296
Selectivity (%)	0.012	0.451
H_2 production rate (mol/h)	0.0001	0.5816

the deactivation of the catalyst, and obtain a steady conversion, selectivity, and hydrogen production rate as same as the start of run values versus MOS.

For the target plant, it was observed that at starting point (MOS=20 min, CO2F=0.053, and H2F=0.213) by setting the decision variables i.e. TR and W/F at 508.1°C and 0.0071, respectively, the observed values for Conv, Sel and FH2 were 4.6%, 40.9%, and 2.143 $mol.h^{-1}$, respectively.

Figure 6 illustrates the output variables of the MSR reactions versus MOS. As seen from this figure, by optimizing the decision variables, it is possible to gain a constant conversion, selectivity and hydrogen production rate for the target process up to 2400 min. By referring to Fig. 3, it can be concluded that after that, the activity of the catalyst falls below 0.73, and manipulating those variables cannot neutralize the deactivation of the catalyst. Therefore, the catalyst should be regenerated or replaced after 2400 min from starting up the MSR process.

Figure 7 shows the optimum values of TR and W/F versus MOS. As expected, when the other operational parameters are constant, the temperature of the reactor should be raised versus MOS to compensate the deactivation of the catalyst. But, after 1700 min,

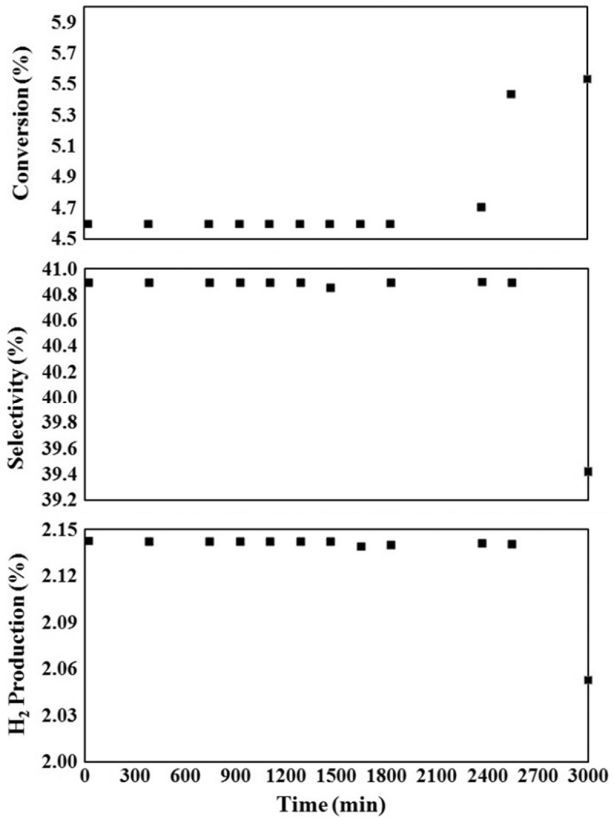

Fig. 6 — Optimum process variables obtained using the HANN model vs. time on stream

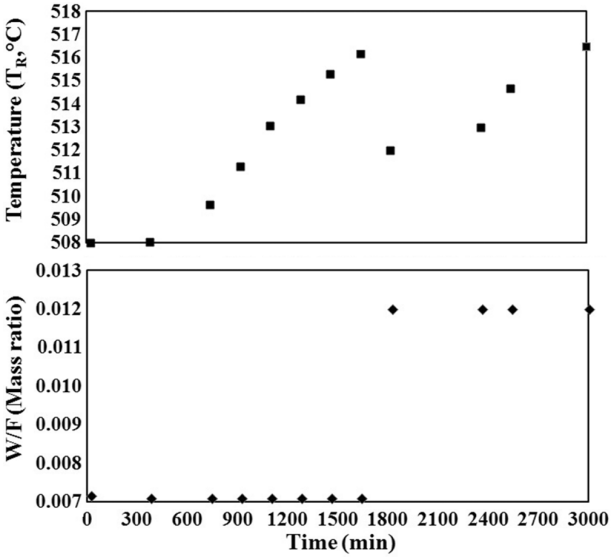

Fig.7 — Optimum selectivity, conversion and hydrogen production vs. time on stream

increasing the temperature of the reactor have possibly reverse effect on the reaction rate due to accelerating the deactivation of the MSR catalyst. Therefore, it is recommended to increasing W/F, and decreasing the temperature of the reactor. Then,

raising the temperature can be followed up to the end of run (MOS=2400 min).

Conclusion

A hybrid decay function-artificial feed forward neural network (HANN) was proposed for a laboratory scale MSR reactor. This model was trained, tested and validated on the basis of actual data obtained from the laboratory experiments. It was showed that the studied commercial $Cu/ZnO/Al_2O_3–ZrO_2$ lost about 31 percent of its initial activity after 3000 min in operation. Moreover, it was confirmed that the proposed hybrid model could predict conversion, selectivity and hydrogen production rate of MSR reaction with the AAD% (average absolute deviation) of 1.296, 0.451 and 0.5816%, respectively. Additionally, the MSE of prediction for these variables were 0.2042, 0.012 and 0.0002, respectively.

After validating HANN, the decision variables of the MSR reaction i.e. reaction temperature and water to feed ratio were optimized such that the deactivation of catalyst could be compensated during the reaction (about 3000 min). Results confirmed that by selecting the optimal values, conversion, selectivity and rate of produced hydrogen could be maintained as equal as the start of run values up to MOS of 2400 min (about 5%, 40.5%, and 2.1 mol/h, respectively). After this point, the process should be stopped, and to meet the desired outputs, the catalyst should be regenerated or replaced. This means that the presented approach was valid to satisfactorily monitor the activity of catalyst, predict the outputs and optimize the outputs of the target MSR plant.

Symbols Used

a	[-]	Activation function
a_c	[-]	Activity of the catalyst
b	[-]	Bias of an ANN node
Conv	[%]	Conversion
CO2F	[-]	Mass fraction of CO_2 in feed
FH2	[mol.h^{-1}]	Molar flowrate of H_2 product
H2F	[-]	Mass fraction of H_2 in feed
m_f	[kg/h]	Mass flowrate of feed
MOS	[min]	Minutes on stream
MSR	[-]	Methanol steam reforming
N	[-]	Total number of data points
P	[Pa]	Inlet pressure of the isomerization reactor
Sel	[%]	Selectivity of methanol reforming reaction
t_c	[h]	Accumulated feed
t_s	[h]	Time interval
TR	[°C]	Temperature of methanol steam reforming reactor
W_{cat}	[kg]	Total weight of the catalyst
x	[-]	Input unit of a neuron
Y	[-]	Output of a neuron in the output layer
z	[m^2]	Final output of a neuron
α	[h^{-1}]	Decay constant

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